

# FCS08 - SOP for Operating and Maintaining Fourier Transform Infrared Spectroscopy (FT-IR) Instruments

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## 1. Scope

- 1.1. This document establishes the procedures for operation, preventative maintenance, and quality control that apply to instrumental analysis of Infrared Spectroscopy (FT-IR). The purpose of these maintenance and quality control (QC) procedures is to ensure that instruments are working properly and are free of contaminants before processing casework.

## 2. Background

- 2.1. To establish a procedure for routine and preventative instrument maintenance and QC to ensure quality and accuracy of reported casework results.

## 3. Safety

- 3.1. Reagent Toxicity:

- 3.1.1. Personnel should refer to the appropriate SDS for solvents and reagents used during analysis for any specific safety requirements.

- 3.1.2. For a complete review of required Health and Safety regulations of the Forensic Science Laboratory (FSL), see *DOM13 - DFS Health and Safety Manual*.

- 3.2. Protective Equipment:

- 3.2.1. Personnel should wear personal protective equipment (PPE) including: lab coat, gloves, and safety goggles when carrying out standard operating procedures.
- 3.2.2. Wear vinyl or nitrile gloves when handling these chemicals to prevent absorption through the skin. If any chemicals are spilled onto gloves, discard gloves into hazardous waste.
- 3.3. Training:
  - 3.3.1. Formal training and authorization in use of instruments and software is necessary.
- 3.4. Personal Hygiene:
  - 3.4.1. Universal Precautions must be followed. Care should be taken when handling instrument, chemicals or any biological specimen. Routine use of gloves and proper hand washing should be practiced.
  - 3.4.2. Refer to *DOM13 – DFS Health and Safety Manual*.
- 3.5. Disposal of Waste:
  - 3.5.1. Waste materials must be disposed of in compliance with laboratory, Federal, state, and local regulations. Solvents and reagents should always be disposed of in an appropriate container clearly marked for waste products and temporarily stored in a chemical fume hood.
  - 3.5.2. Consult DFS Safety Officer for proper procedures (See *DOM13 – Health & Safety Manual*).

## 4. Materials Required

- 4.1. Reagent Grade Methanol (MeOH) or better
- 4.2. Polystyrene and Caffeine or Procaine Standards
- 4.3. Binder/Folder for Standard results, or electronic equivalent
- 4.4. Quarterly/Weekly Maintenance Logbook, or electronic equivalent

## 5. Standards and Controls

- 5.1. Polystyrene Standard (Thermo Scientific, VALPRO ATR Standard Set 840166700, or equivalent)
- 5.2. Procaine Standard (VWR, TCP1961-25G, or equivalent)
- 5.3. Caffeine Standard (VWR, BT127310-100G, or equivalent)

## 6. Calibration

6.1. Not applicable

## 7. Procedures

### 7.1. Performance Verification of Instruments

7.1.1. Prior to being used in casework, newly installed FT-IR instruments shall undergo a performance verification to ensure it meets manufacturer's specifications and that results are produced as expected.

7.1.2. If an instrument is moved from its original location, a performance verification must be performed again prior to resuming casework on the instrument.

7.1.2.1. Note: The Agilent Cary 630's compact design was made for easy portability. Movement of this instrument does not require a performance verification.

7.1.3. Performance verifications shall consist of the following:

#### 7.1.3.1. Wavelength accuracy study

7.1.3.1.1. Using a validated method, acquire a procaine spectrum five times a day for three consecutive days.

7.1.3.1.2. Acceptance criteria: absorption bands 3203  $\text{cm}^{-1}$ , 2584  $\text{cm}^{-1}$ , and 1691  $\text{cm}^{-1}$  all within 5  $\text{cm}^{-1}$ .

#### 7.1.3.2. Spectral Accuracy study

7.1.3.2.1. Using a validated method, acquire spectra for ten drug standards.

7.1.3.2.2. Acceptance criteria: Comparison of acquired spectra to verified reference spectra must meet criteria outlined in Table 1.

### 7.2. Weekly Maintenance Procedure

7.2.1. Weekly Maintenance must be carried out every week on the instrument used for casework and must be performed prior to any other casework. If only one instrument is used for casework, only the instrument being used shall undergo this weekly maintenance. Weekly maintenance must be performed only if the instrument is in use that week.

7.2.2. Monday is considered the start of a new week for weekly maintenance purposes.

7.2.3. Instrument Ready Check (for **Perkin Elmer Spectrum 2** only)

7.2.3.1. Clean instrument using methanol prior to initiating the Ready check

7.2.3.2. Select Run Selected from the top panel (top right). Click Scan.

7.2.3.3. Print report and file in the FT-IR Logbook.

7.2.4. Diagnostic Values (for **Agilent Cary 630** only)

7.2.4.1. View diagnostic values on the Diagnostics page of the software

7.2.4.2. Ensure values are within Marginal value at minimum (Optimal values preferred).

7.2.4.3. Print report and file in the FT-IR Logbook.

7.2.5. Performance Check

7.2.5.1. Run a negative control (blank) followed by positive control (procaine standard) using the sample run procedure as outlined in section 7.7.

7.2.5.2. If the procaine standard matches the standard in the search library, print the search results using the report template and file it along with the corresponding blank in the FT-IR Logbook.

7.2.5.3. Another substance may be used for this procedure upon approval from the Technical Lead and shall be documented on the maintenance logbook.

7.3. Quarterly Maintenance Procedure

7.3.1. Quarterly Maintenance shall be carried out every three months on the instrument used for casework and must be performed prior to any other casework. If only one instrument is used for casework, only the instrument being used shall undergo this quarterly maintenance. Quarterly maintenance must be performed only if the instrument is in use during the covered time period.

7.3.2. Quarterly maintenance for the **ThermoFisher Nicolet iS50** includes Suitability Checks and a Val Pro Qualification.

7.3.2.1. Suitability Check using Polystyrene

7.3.2.1.1. Clean instrument using methanol prior to initiating the Polystyrene Check.

7.3.2.1.2. Select Polystyrene Check from the Experiment pull-down menu.

7.3.2.1.3. Select the System Status. Then select System Suitability. Then select Run.

- 7.3.2.1.4. Follow the screen prompts to collect a background, followed by a Polystyrene standard.
- 7.3.2.1.5. Pop-up window will appear once completed. Select Report.
- 7.3.2.1.6. If the Polystyrene check passed, print Report and file in the FT-IR Logbook.
- 7.3.2.2. Suitability Check Using Caffeine or Procaine Hydrochloride (whichever is used)
  - 7.3.2.2.1. Clean the ATR using methanol prior to initiating the Caffeine or Procaine Check.
  - 7.3.2.2.2. Select Caffeine or Procaine Check from the Experiment pull-down menu.
  - 7.3.2.2.3. Select the System Status. Then select System Suitability. Then select Run.
  - 7.3.2.2.4. Follow the screen prompts to collect a background, followed by the Caffeine or Procaine standard.
  - 7.3.2.2.5. Pop-up window will appear once completed. Select Report.
  - 7.3.2.2.6. If the check passed, print Report and file in the FT-IR Logbook.
- 7.3.2.3. Val Pro Qualification
  - 7.3.2.3.1. Wipe instrument plate with methanol prior to initiating the Val Pro qualification.
  - 7.3.2.3.2. Select Analyze on the top toolbar. Then Click on Val Pro Qualification
  - 7.3.2.3.3. Select a Qualification test from the pull down menu: Nicolet iS50 system with integrated ATR and built-in DTGS-EP
  - 7.3.2.3.4. Follow the screen prompts to collect a background, followed by the Polystyrene standard.
  - 7.3.2.3.5. If qualification failed, clean instrument and polystyrene accessory and restart the Val Pro Qualification.
  - 7.3.2.3.6. Print Report and file into the FT-IR Logbook.
- 7.3.3. Quarterly maintenance for the **Perkin Elmer Spectrum Two** includes an Instrument Verification.
  - 7.3.3.1. Clean instrument analysis surfaces (ATR) using methanol prior to initiating Instrument Verification check.

7.3.3.2. Select Instrument Verification from the top panel (top right).

7.3.3.3. Click Scan.

7.3.3.4. If the Instrument Verification passed, print report and file in the FT-IR logbook.

7.3.4. Quarterly maintenance for the **Agilent Cary 630** includes a Performance Test, Stability Test, and Laser Frequency Calibration Test.

7.3.4.1. Clean the ATR using methanol prior to initiating each check.

7.3.4.2. Run each test using the Advanced Features » System Check window and follow screen prompts.

7.3.4.3. If checks passed, print reports for each test and file in the FT-IR Logbook.

#### 7.4. As-Needed Maintenance Procedures

7.4.1. A performance check must also be completed after any major maintenance or event that may modify the function of the equipment (i.e., changing of major internal parts).

7.4.2. Replace dessicant when humidity level reaches 30%, or when specified by indicator.

#### 7.5. Preventative Maintenance Schedule

7.5.1. Preventative maintenance shall be performed by the supplier as per their recommended schedule (annually, i.e., once per calendar year).

#### 7.6. Control Chart and Logbook Maintenance

7.6.1. As appropriate, the significant parameters appropriate for the identification of procaine (or other substance used for quality control) shall be recorded in the laboratory control chart for FT-IR. Critical pieces of information include peak transmittance and position of characteristic peaks.

7.6.2. An FT-IR maintenance log (Document Control Number 30398) shall be maintained for each instrument to reflect any maintenance procedures performed (along with corresponding dates and analysts) and operating status (i.e. online or offline).

#### 7.7. Sample Run Procedure

7.7.1. Ensure the sample window is clear on the Attenuated Total Reflectance (ATR).

7.7.2. Ensure the 16 Scans experiment (or other validated method) is selected.

- 7.7.3. Collect a background spectrum without lowering the anvil. Note: background spectrum does not need to be saved.
- 7.7.4. Collect a blank spectrum with the anvil lowered. Name the run with identifying information, save, and print using the blank report template.
- 7.7.5. Place appropriate amount of sample directly on the ATR sample window. Collect spectrum, name the run with identifying information, and save.
- 7.7.6. Search the standard library for an appropriate match against acceptance criteria outlined in Table 1.
  - 7.7.6.1. Note: For Spectrum Two FT-IR, sample collection and library search can be performed simultaneously using the “Scanalyze (Scan and Search)” button
- 7.7.7. Print the search results using the report template and retain it with casework along with the blank.
- 7.7.8. Perform additional extraction and rerun the sample, if desired.
- 7.8. Quality Control Procedures
  - 7.8.1. A background spectrum shall be obtained immediately prior to each sample.
  - 7.8.2. A negative control (blank run) shall be run prior to each sample using the same parameters as the sample analysis. The blank must be free of discernible peaks to be acceptable.
  - 7.8.3. Sample run printouts shall be accompanied by the previous blank run’s printout to ensure the system was free of contaminants prior to the run of interest.
  - 7.8.4. Only one sample shall be run on FT-IR at a time to ensure that the correct sample is run and to avoid contamination.
  - 7.8.5. The ATR diamond and anvil shall be wiped clean with methanol (or other equivalent cleaning solvent) after each sample or standard run.
  - 7.8.6. Sample acceptance Criteria
    - 7.8.6.1. Performance criteria are assessed using the following acceptance criteria and interpretation parameters:
      - 7.8.6.1.1. An unknown spectrum is suitable for comparison to a known when there are visible peaks present.
      - 7.8.6.1.2. Library searches can be used to provide useful information pertaining to the identity of a compound but should not be used as a replacement for analyst verification of the overall appearance and the presence and location of major spectral peaks

when making an identification.

7.8.6.1.3. If used for identification, results from library searches must be printed and retained with sample spectra.

7.8.6.1.4. The infrared spectrum of the majority of controlled substances and other substances routinely identified is specific to a single compound and may be used for structural identification.

**Table 1.** Fourier Transform Infrared Spectroscopy (FT-IR) Acceptance Criteria

<b>FT-IR PARAMETERS</b>	<b>Acceptance Criteria</b>	<b>Detail</b>
	Spectral Pattern	Overall spectral pattern shall be consistent with reference standard, as evaluated by the analyst (relative peak intensity ideally within 20%)
	Position of major peaks	As evaluated by the analyst; ideally within 5cm <sup>-1</sup> .
	Peak width match	As evaluated by the analyst; ideally within 20%.

## 7.9. Standard Library Searches

7.9.1. Verified standards shall be added to the FT-IR Standards Library as they are received, and their spectra shall be used as the basis for a successful match.

7.9.2. The analyst shall confirm a library match through visual inspection of the sample spectrum against the standard spectrum.

7.9.3. To be conclusively identified, a sample spectrum must match a verified standard spectrum and must pass a visual inspection against the standard spectrum.

7.9.4. A copy of the matching search results and the blank run immediately before it shall be included with the printout of the sample.

7.9.5. All data shall be saved in order to be reviewed later.

## 8. Sampling



8.1. Not applicable

## **9. Calculations**

9.1. Not applicable

## **10. Uncertainty of Measurement**

10.1. Not applicable

## **11. Limitations**

11.1. Not applicable

## **12. Documentation**

12.1. Maintenance Logbooks (Document Control Number 30398) and Control Charts

## **13. References**

13.1. DFS Departmental Operations Manuals (current revisions).

13.2. Forensic Chemistry Unit SOPs (current revisions).

13.3. Controlled Substances Standard Operating Procedures; Comparative and Analytical Division, Houston Forensic Science Center (Document ID 2923, July 3, 2017)